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Danish Chinese Center for Nanometals - Annual report 2012

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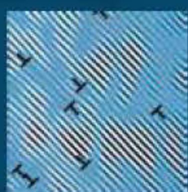
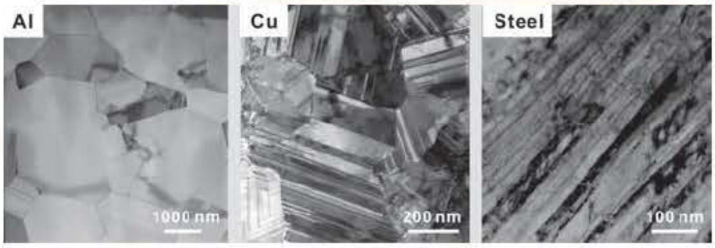


Danish – Chinese Center for Nanometals

Nanometals

The Center investigates metals, including alloys, with internal length scales ranging from a few nanometers to a few micrometers. These are termed nanometals, and have new and interesting properties, such as exceptional mechanical strength, which are related to their internal structure. The scientific focus is to understand and control the mechanisms and parameters determining the mechanical and physical properties of such nanometals as well as their thermal stability.

The internal structure of nanometals is highly diverse, consisting of different types of internal interfaces, spatially arranged in different morphologies, as exemplified by equiaxed grains in aluminum, lamellar nanotwins within equiaxed sub-micrometer grains in copper, and lamellar boundaries between two phases in steel. The morphology as well as the chemical composition of nanometals can be manipulated and are key features to be optimized for future applications of nanometals.



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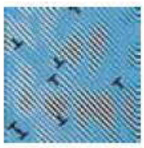


Danish – Chinese Center for Nanometals

Introduction

At the end of 2011 we received the good news that our application for a three-year extension of the Center (until August 2015) was approved by the Danish National Research Foundation, supplementing an approval in China by the National Nature Science Foundation of China. With funding secured, and by the hiring of three young, talented materials researchers, the Center has been able to make a fast start in the new period. This is demonstrated in the present report for 2012 covering work belonging to both the first and the new Center period, and which shows a smooth transition, not affected significantly by the organizational changes within DTU in 2012. The report describes both scientific and technical highlights, as well as publications and international collaboration. Achievements in these areas reflect the initial vision of the Danish National Research Foundation that the Center should create both synergy and complementarity in a joint Danish-Chinese collaboration. Active dissemination of knowledge acquired within the Center is also part of this vision. In 2012 this area has been addressed through organization of an international workshop and in the arrangement of the 33rd Risø International Symposium on Materials Science.

The achievements summarized in the report also reflect the current national and international trend that besides being on a high scientific level, research results also should be evaluated with a view to their application in society. This is a difficult balance to achieve, but we try by presenting an exam-

33rd Risø International Symposium on Materials Science

ple to demonstrate how our fundamental research can form nuclei for more applied projects. The example given is, for good reason, inspired by the fact that the Center is now part of the Wind Energy Department of DTU, which focusses on optimization and durability of wind turbines both on land and offshore. A very important area is the durability of mechanical components in wind turbines, where friction, wear, and fracture of gears and bearings must be better understood in order to improve the economics of wind power. Here our research on strong, fatigue and wear resistant surface layers induced by plastic deformation creates knowledge that is almost directly applicable in materials development and component design. Such nanostructured layers can be produced by plastic deformation as reported in highlights No. 2 and No. 3. These highlights also demonstrate the high degree of synergy and complementarity between the work in China and in Denmark.

An important area within the Center is the development and application of advanced characterization techniques. Techniques given high priority are focused on electron microscopy, where we have expanded our line of transmission electron microscopes with a new TEM 2100 thanks to a grant from the Villum Kann Rasmussen Foundation. The research and development in the area of transmission electron microscopy has led to the demonstration of a non-destructive 3D orientation mapping technique with a very high spatial resolution of down to 1 nanometer. The team behind the development, which includes senior scientist Dr. Xiaoxu Huang from the Center, received in 2012 the Microscopy Today 2012 Innovation Award for their technique called 3D OMiTEM.

A new initiative to strengthen the scientific competences within the Center is the arrangement of very focused workshops, with participation of leading international scientists with skills complementary to those of the Center scientists. The first workshop of this kind was held from 27th – 31st August on the subject of boundary migration. The workshop was very inspiring and created many promising contacts between the participants, representing advanced materials characterization and theoretical modeling from the Center, and groups in USA and Belgium with expertise in atomistic and mesoscale modeling and simulation (see highlight No. 6).

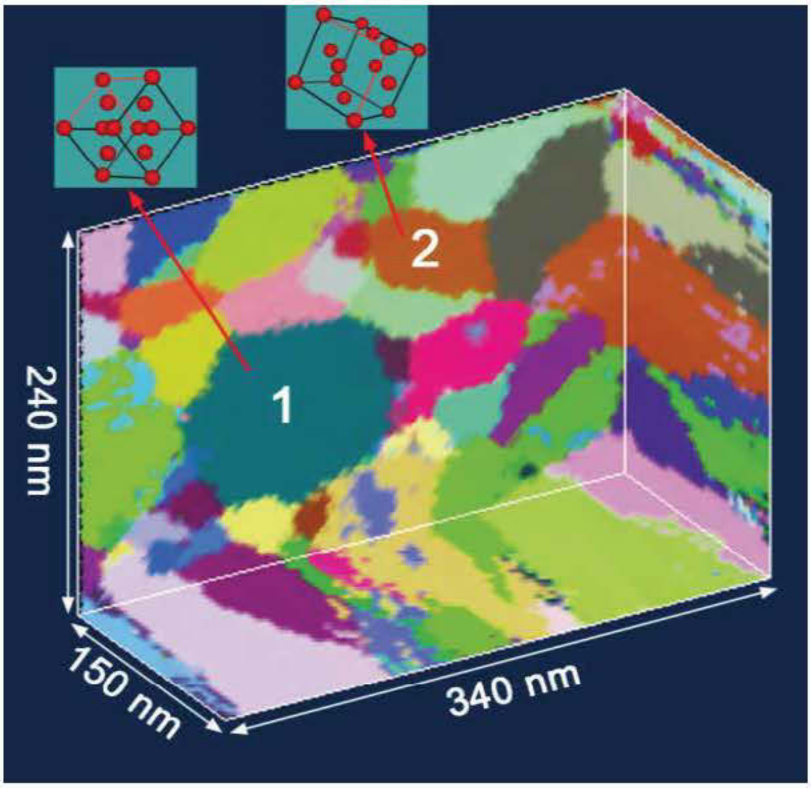
As mentioned above, the Center also arranged the 33rd Risø International Symposium, entitled "Nanometals – Status and Perspectives" held from 3rd-7th September 2012 at DTU, Risø Campus. In accordance with the traditions of the Risø Symposia, invited papers (12) and contributed papers (31) addressed important current problems to illustrate global research and development in the field of nanometals from theory to application. As a new initiative a special session was also held where 4 speakers introduced and discussed international R&D, including funding, in the area of nanostructured materials in USA, China and Japan.

The Risø Symposium gave ample opportunity for the senior and junior members of the Center to discuss ongoing work, and for the senior scientists to discuss in detail future activities as well as administrative matters. In addition, several members of the Center met at two workshops on themes complementary to those of the Center. One was on Bulk Nanostructured Metals on 27th-29th June at Kyoto University, Japan, organized by Professor N. Tsuji, and the other on Advanced Structural Materials, held on 14th-15th November at Chongqing University, China, and organized by Dr. X. Huang. These two workshops combined international and national lecturers with an audience predominantly composed of teachers and students from Japanese and Chinese universities. So in summary, the year has been very successful in outreach activities, which have broadened significantly the international knowledge and recognition of the Center activities.

In conclusion, the Danish-Chinese Center on Nanometals is judged to be in very good shape, due both to the results obtained and to the strong interest in center activities shown by all participants. Good governance, a high level of science, and teamwork and friendship characterize the Center, and we look forward with optimism to the coming years. Here an important objective is to bring our knowledge, especially on processing and characterization of nanometals, closer to application thereby to benefit both the Danish and the Chinese societies now and in the future.

Nanoscale analysis of materials structures in 3D

The use of high energy X-rays for the 3D structural analysis of metals and alloys has been an important area of development at Risø for the last 10-15 years, supported to a great extent by the Danish Natural Research Foundation. The developments have led to a reduction in the spatial resolution of the techniques to below one micrometer, defining a future goal for futher development of x-ray techniques. To reach even finer structural scales, in a collaboration between DTU, Risø campus, DTU Physics, Tsinghua University, IMR, SYNL, and John Hopkins University, a new electron microscope-based technique has been developed that allows non-destructive orientation mapping with a spatial resolution of 1 nm, which has been reached in an exploratory investigation of nanostructured aluminium. The requirement of the new technique led to new specifications for an advanced electron microscope, which has now been procured, thanks to a generous grant from the Villum Kann Rasmussen Foundation.

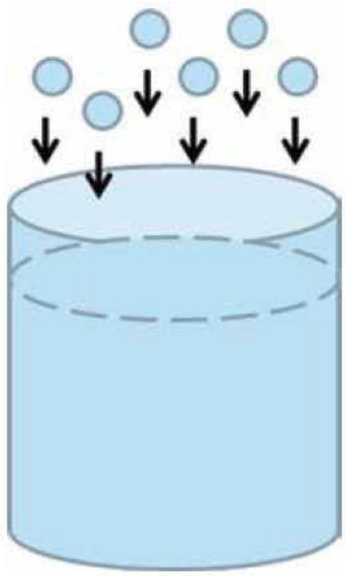


3D grain orientation map from part of a nanocrystalline aluminum sample

The new microscope in operation

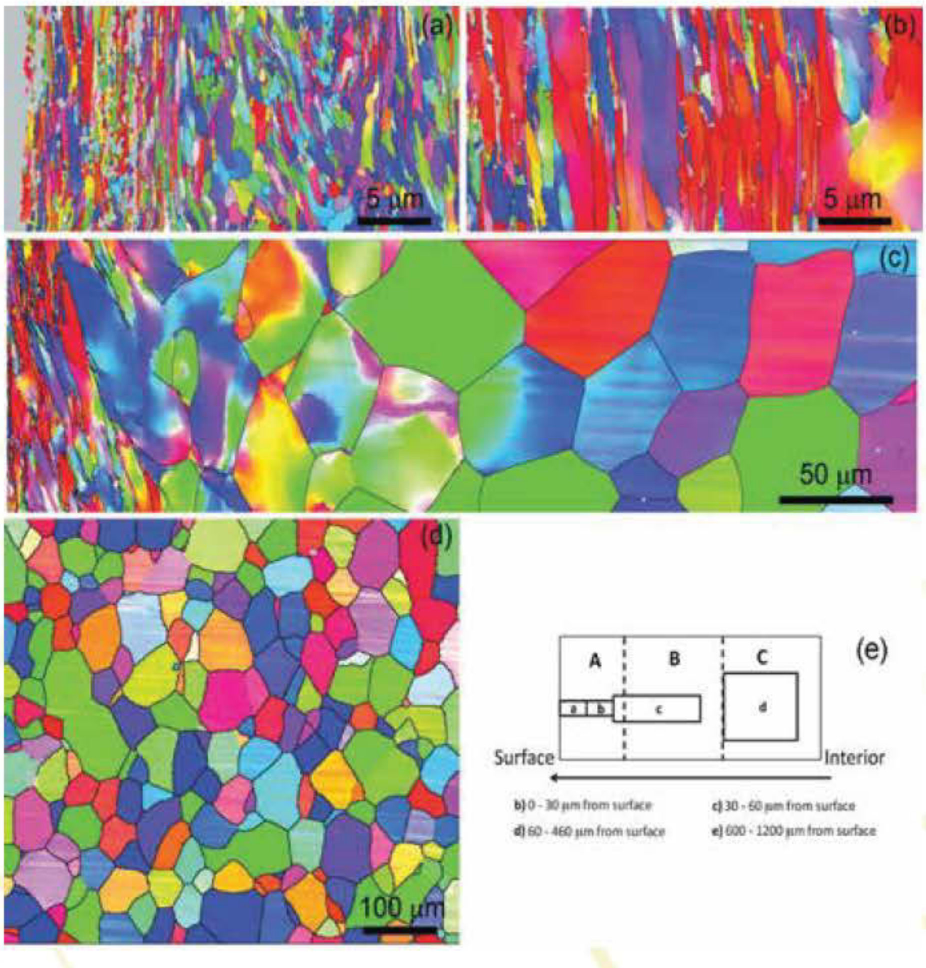
Hard surfaces in steel plastically deformed to high strains

Metal surfaces in components for wind turbines such as gears and bearings are plastically deformed during operation, leading to crack formation and failure. The deformed surfaces and subsurface layers are on the nanoscale and their description requires advanced structural and mechanical characterization techniques. In steel samples, a hard surface layer has been introduced by particle bombardment (see upper figure) by use of a shot peening (SP) process. The resulting deformation microstructure is on the nanometer scale, down to 50 nm near the surface, with a flow stress of about 800 MPa in the subsurface layer about 25 μm from the surface. This is 3-4 times higher than the flow stress of the steel before the shot-peening process is applied. The surface and subsurface structures have been analyzed quantitatively by electron microscopy, and based on a theoretical model the flow stress just below the surface has been estimated to be as high as 1400 MPa. In general, the characterization of the surface and subsurface layers will form the basis for the formulation of constitutive equations, which are important for use in finite element modeling of friction and wear of industrial components.



Plastic deformation by particle bombardment. (See also the following highlight where SMAT is applied.)

Microstructure at different positions from the surface for the shot-peened sample, characterized by electron backscatter diffraction (EBSD).

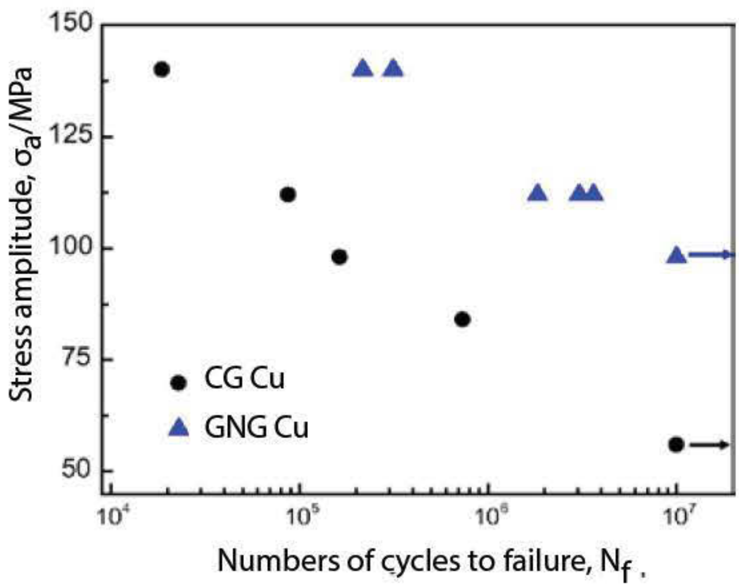


Enhanced fatigue properties in gradient nano-grained copper

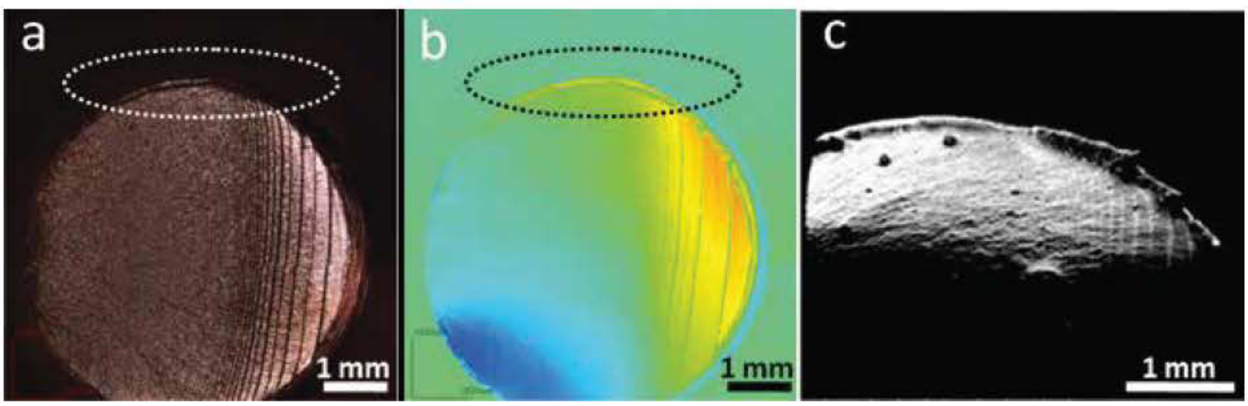
Most fatigue failures occur at material surfaces and then propagate into the interior. Thus, an architectured structure with a nanostructured surface layer and a coarse-grained (CG) interior may enhance the fatigue resistance of a metal. A strong nanostructured surface layer may suppress fatigue crack initiation, and crack propagation may be arrested by a CG interior once a crack has nucleated at the surface.

To test these ideas gradient nano-grained (GNG) Cu samples with a nanostructured surface layer of about 50 μm thick have been prepared by surface mechanical attrition treatment (SMAT) at IMR, SYNL. The fatigue strength of the GNG-Cu samples, which is about 98 MPa based on a fatigue life of 10^7 cycles, is raised by about 75% compared with the fatigue strength of CG-Cu (~56 MPa). The improvement in fatigue life of GNG-Cu during low cycle fatigue tests is even more impressive, with a fatigue life about 14 times longer than that of CG-Cu at a stress of 140 MPa. The improved fatigue strength and fatigue life of GNG-Cu are attributed to the GNG surface layer, which not only contributes to the enhanced fatigue strength but also suppresses crack nucleation. The cyclic deformation of GNG-Cu induces an abnormal grain coarsening, which initiates from the sub-surface layer and grows along a region 45° to the stress axis toward the top of the surface layer, where the fatigue cracks start to form.

Dependence of the fatigue life (Nf) on the cyclic maximum stress for coarse-grained (CG) Cu and gradient nano-grained (GNG) Cu.



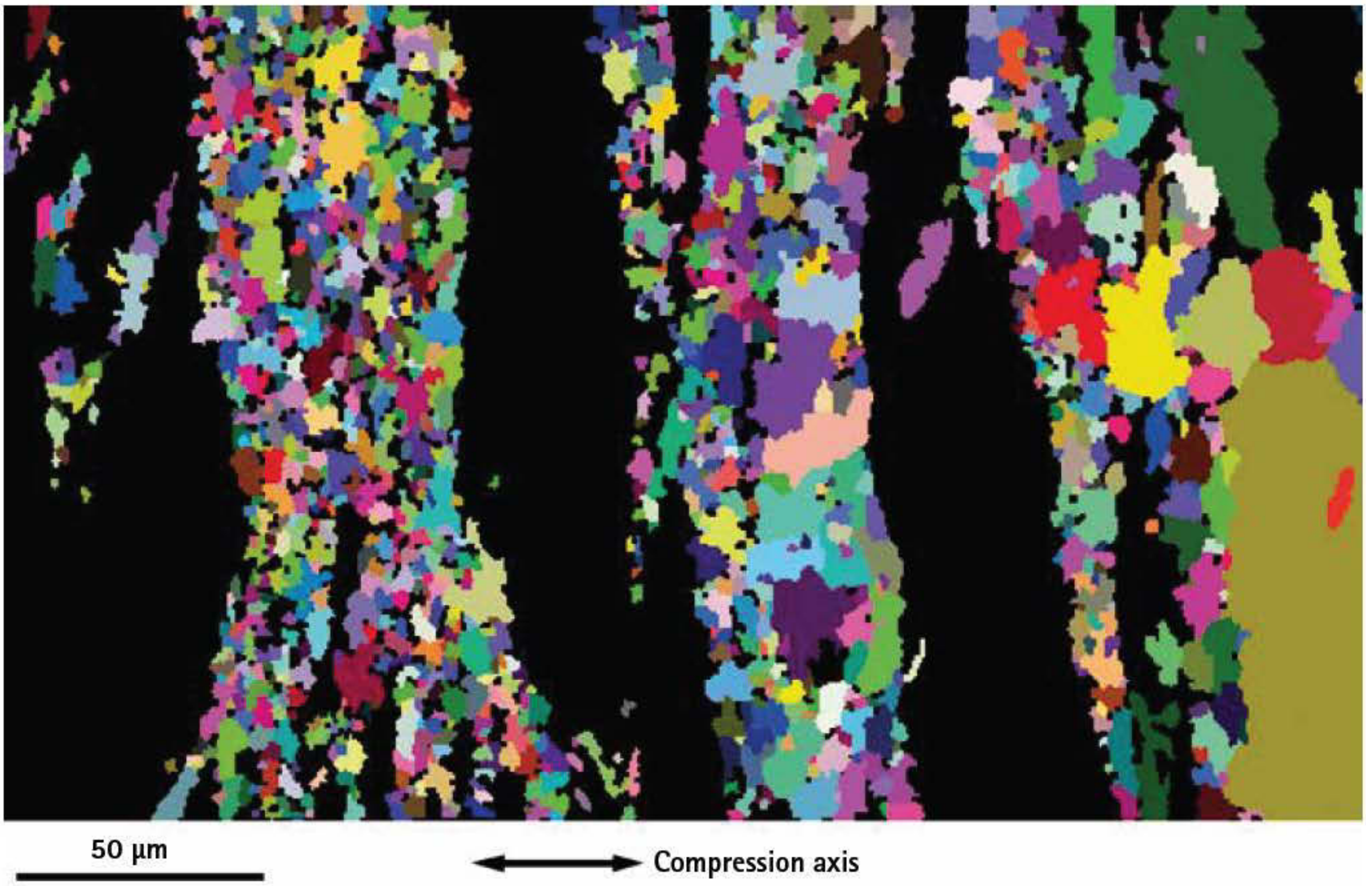
Fracture surface of a fatigued GNG Cu sample observed by (a) SEM and (b) laser confocal microscopy; (c) a high-magnification image of the area in (a).



Recrystallization kinetics in nanocrystalline copper

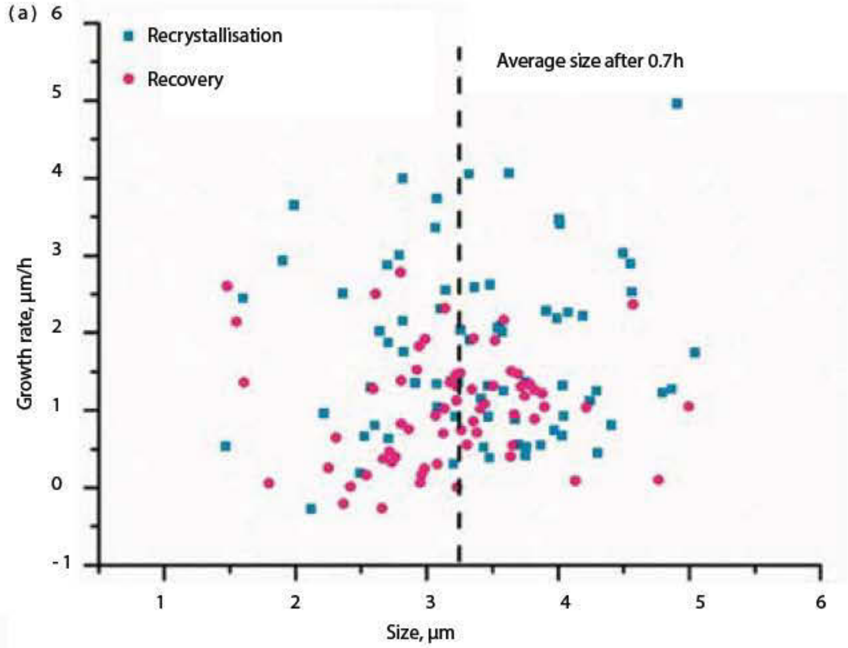
In a collaboration between DTU Risø Campus and IMR, Shenyang, the recrystallization kinetics in copper deformed at liquid nitrogen temperature by dynamic plastic deformation (DPD) to a strain of 2 has been investigated. This was to explore earlier observations that the uniform elongation of the DPD sample is poor and improvement can only be obtained if the sample is annealed to 80% recrystallization. The new investigation showed that the deformed microstructure is very inhomogeneous with large regions of low stored energy and other regions heavily subdivided by twins and dislocation boundaries, and thus of a much higher stored energy. As a consequence the recrystallization is very inhomogeneous, resulting in large regions which are fully recrystallized intermixed with large unrecrystallized regions (See the figure below). As a cure to this problem, DPD samples have been additionally cold rolled in order to homogenize the deformed microstructure. In these samples, slight annealing leads to a significant increase in uniform elongation and thus also in the applicability of the material.

Partly recrystallized microstructure of a DPD sample annealed at 120°C for 1 h. The recrystallized grains are shown in random colors and the non-recrystallized regions are shown in black.



In-situ synchrotron x-ray characterization of annealing kinetics in nanostructured aluminium

In a collaboration between Chongqing University and DTU, Risø Campus, the coarsening, nucleation, and growth of individual bulk crystallites have been determined by in-situ non-destructive three-dimensional x-ray diffraction (3DXRD). The material investigated was aluminium (AA 1200) cold rolled to a strain of 4 (98.2% reduction in thickness). It was found that initially during the annealing, coarsening by recovery dominates. Later, recrystallization starts, and by fast growth of recrystallization nuclei, some very big grains evolve. This occurs simultaneously with coarsening by recovery in other parts of the microstructure. This is in agreement with previous results for other materials investigated within the Center. The new 3DXRD experiment has shown that the nuclei (the crystallites that end up being very large by fast growth) do not need to have an initial size advantage compared to those that coarsen at a much lower rate by recovery; i.e. conventional grain growth cannot explain the present results. Data of this type are considered very important for understanding the thermal response of nanometals, and can help to design guidelines for thermal treatments of nanostructured metals in order to optimize mechanical properties with a view to applications.



Distribution of growth rates as a function of crystallite size at an early stage of annealing of aluminium cold rolled to a strain of 4. All the "blue" crystallites (data points) are those that grow to become bigger than the average size after annealing for 20 h at 257°C, whereas the "red" crystallites end up having a size below the average. It is seen that many blue crystallites are among the smallest at this early annealing stage.

In-situ synchrotron experiments were carried out at the European Synchrotron Radiation Facility (ESRF), Grenoble, France.

International "Work-workshop" on boundary migration

As a new idea it was decided to bring together a small group of internationally leading experts within a strongly focused topic – boundary migration – and create a week-long discussion forum, where the newest experimental and modeling results could be discussed in detail and new ideas and in-depth understandings could be stimulated. The external participants were selected with particular focus on modeling experts to supplement our own expertise:

- *Elizabeth A. Holm, Sandia National Laboratories, USA (now Carnegie Mellon University, USA) – Mesoscale modeling*
- *Nele Moelans, Leuven University, Belgium – Phase Field simulations*
- *Stephen M. Foiles, Sandia National Laboratories, USA – MD simulations*

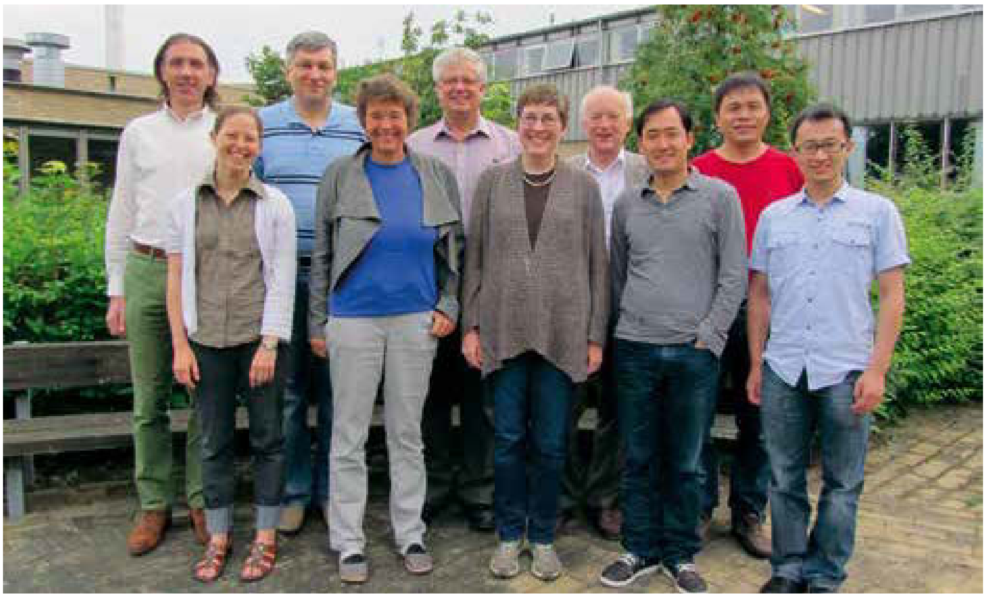
From the Center:

- *Andy Godfrey – Experiments and Phase Field simulations*
- *Dorte Juul Jensen – Recrystallization in general*
- *Hongwang Zhang - Plastic deformation and microstructure*
- *Niels Hansen – Microstructure and mechanical properties in general*
- *Oleg Mishin – Texture effects on recovery and recrystallization*
- *Tianbo Yu – Electron microscopy on recovery*
- *Xiaodan Zhang – Microstructure and mechanical properties on the nanoscale*
- *Yubin Zhang – Synchrotron experiments on recrystallization*

Prior to the start of the the "work-workshop" each participant selected their newest, most relevant, already published work and these papers were distributed among all participants. At the "work-workshop" these results were put into perspective, including further information on the assumptions, uncertainties etc. involved, plus of course new, still unpublished results were presented and discussed. Each day had a specific theme and began with short oral presentations, after which the rest of the day was spent on discussions, some in plenum and some in smaller groups. The "work-workshop" was held at Risø, and the participants had offices near to each other so that discussions could easily be continued whenever needed.

The "work-workshop" week turned out to be an outstanding and very intense week. Many new ideas for further analysis and new experiments and modeling possibilities were conceived, some of which are already being used in preparation of publications, and some of which are being followed up by individual and collaborative projects. Just to mention one scientific aspect: the "work-workshop" highlighted the importance of mobility, and the focus of discussions was on the possibility that even boundaries with almost identical misorientations may have quite different mobilities.

All participants agreed that the "work-workshop" concept was very successful and this format will be used again for another scientific topic.



Public Outreach

- Participation in the European Wind Energy Association (EWEA) 2012 Annual Event, April 16th – 19th, Bella Center, Copenhagen. Structural characterization by electron microscopy of steel components of wind turbines was introduced and demonstrated to a large number of visitors.

- Visit to LORC (Lindoe Offshore Renewables Center) on May 2th with a presentation of the work within the center followed by networking.

- Organization of the 33th Risø International Symposium on Materials Science entitled "Nanometals – Status and Perspectives", September 3th-7th, DTU, Risø Campus, with 59 participants from 11 different countries. The symposium is an important international scientific and technical event with substantial financial support from the following foundations: Civilingeniør Frederik Leth Christiansens Almennyttige Fond, Danish National Research Foundation, Fabrikant Mads Clausens Fond, Knud Højgaards Fond, Kraks Fond and Otto Mønsteds Fond.

Visit to LORC

Exchange

Exchanges of scientists are specified below. As before, such exchanges are supplemented with preparation and circulation of extensive quarterly progress reports. In combination, Center participants in Denmark and China are always well informed and the research and development can progress without unnecessary delay.

Visits from Tsinghua to DTU Risø campus

Prof. Andy Godfrey
PhD student Guomin Le
PhD student Haiyan Xu

Visits from IMR SYNL to DTU Risø campus

Prof. Ke Lu
Prof. Lei Lu
Dr. Hongwang Zhang
Dr. Ming Cheng

Visits from Chongqing University to DTU Risø campus

Prof. Qing Liu
Dr. Guilin Wu
Dr. Tianlin Huang
Dr. Renlong Xin
Dr. Zhiqing Zhang

Visits from DTU Risø campus to China

Dr. Techn. Dorte Juul Jensen to Tsinghua, IMR SYNL and Chongqing University
Dr. Techn. Niels Hansen to Tsinghua, IMR SYNL and Chongqing University
Senior Scientist Xiaoxu Huang to Chongqing University and IMR SYNL
Dr. Chuanshi Hong to IMR SYNL
PhD student Fengxiang Lin to IMR SYNL

Publications

Journal articles

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Honors and Awards

Dr. Techns. Niels Hansen and Dorte Juul Jensen were awarded the Institute Awards 2012 - Cook/Ablett Award by the Institute of Materials, Minerals and Mining for their paper, 'Deformed metals – Structure, recrystallisation and strength', published in Materials Science and Technology, vol. 27, no. 8, pp. 1229-1240, 2011.

Prof. Lei Lu was awarded the Young Female Scientists Award of China.

PhD student Jacob Kidmose won the silver medal in the Best Oral Presentations by Young Scientists category at the 7th International Symposium on Ultrafine Grained Materials (UFG-VII), as part of the TMS Annual Meeting 2012, Orlando, Florida.

Senior Scientist Xiaoxu Huang, together with Søren Schmidt from DTU Physics and other co-workers, received the Microscopy Today 2012 Innovation Award for development of a non-destructive 3D orientation mapping technique (3D-OMiTEM) with spatial resolution down to 1 nm.

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